



TestAmerica Savannah
5102 LaRoche Avenue
Savannah, GA 31404

Tel: (912) 354-7858
Fax: (912) 352-0165
www.testamericainc.com

January 20, 2012

Rick Wilkin
US EPA National Risk Management Research
919 Kerr Research Drive
Ada, OK 74820
wilkin.rick@epa.gov
Tel: (580) 436-8874

Subject: Request for Proposal
MBAS Testing
Quote Number 68009738

Dear Rick Wilkin:

On behalf of TestAmerica Laboratories, Inc., I am pleased to submit pricing for the MBAS Testing. TestAmerica Savannah is eager to support this commitment by making our personnel and analytical laboratory resources available to you. It will be necessary to communicate with the lab during this sampling to ensure that lab resources are available to meet the short holding time. The quote also contains an additional fee that will be applied to samples that arrive on Saturday.

We thank you for choosing TestAmerica Savannah, and we look forward to working with you on this project. The following quotation contains a detailed price breakdown, as well as any notes and clarifications pertaining to your project. This quotation is subject to TestAmerica's Terms and Conditions, unless otherwise agreed upon in writing. Should you have any further questions or require additional information about our analytical services, please feel free to contact me at 912-354-7858 or via email at the address listed below.

Sincerely,

Bernard Kirkland
Project Manager
bernard.kirkland@testamericainc.com

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Savannah, GA 31404

Prepared for:

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Tel: (580) 436-8874

Prepared by: Kirkland, Bernard
Date: 1/20/2012
Expiration Date: 4/19/2012
Est. Start Date:

Project: MBAS Testing

Quote Number: 68009738 - 0

MBAS

TAT: 10_Days (Business Days)

Matrix	Method	Test Description	Quantity	Unit Price	Extended Price
Water	425.1	Methylene Blue Active Substances (MBAS)	10	\$ 36.27	\$ 362.70
Total MBAS					\$ 362.70

Quote Other Charges

Description	Quantity	Unit Price	Extended Price
Labor - Weekend Coverage	1	\$ 100.00	\$ 100.00
Total Other Charge			\$100.00

Total Other Charges	\$ 100.00
Total Analysis Charges	\$ 362.70
Grand Total for Quote 68009738	\$ 462.70

**Quoted charges do not include sales tax. Applicable sales tax will be added to invoices where required by law.

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PROJECT DETAILS

Coolers and Supplies

TestAmerica will provide high quality sample containers and coolers to support analytical activities. Supplies may include a slight bottle excess (10%) in case of breakage. The cost of bottles and cooler usage is included in the analytical price quoted. TestAmerica expects that all coolers will be returned to a TestAmerica laboratory. TestAmerica further anticipates that approximately 70% or more of the containers supplied will be returned as samples. Coolers not received back by the projected deadline, or as arranged with the PM, may be charged at \$30 per cooler. Similarly, if less than 70% of the containers provided are returned as samples, TestAmerica may charge for additional containers provided at a flat rate of \$1 per container.

Deliverables

Unless a level III or IV deliverable is specifically listed on the pricing page, this quotation only includes delivery of a Level II report. Level III or IV reports may be available at an additional charge.

MS/MSD Samples

TestAmerica complies with the required frequencies for MS/MSD per batch. When MS/MSD are not specifically requested, TestAmerica will strive to perform the required QC using whatever available sample there is but will not report the QC results unless the client requests it specifically. Any requested MS/MSD will be charged at unit rates. If MS/MSD are required/requested, the client must provide additional sample volume.

Multiple Report Formats

TestAmerica will provide two analytical report formats. The option is a hard copy report and a standard TestAmerica EDD or PDF report, or any combination of two of these three choices. The cost of this deliverable approach is included in the analytical cost. Client must specify which deliverables are required. Opting for only one deliverable will not prompt for a discount. Additional report copies or deliverables beyond the above described service can be provided subject to an associated fee. Data that requires retrieval from permanent storage may incur an additional archival charge. Please contact your PM to inquire about availability and cost of additional deliverables.

QC Limit Disclaimer

The laboratory's reporting limits, detection limits, and control limits are subject to change as these values are updated periodically to reflect analytical sensitivity and capability.

Sample Container Shipping

The containers and preservatives required by the project shall be delivered via ground transportation at no additional cost to the client. A minimum of 3-5 days advance notice (depending on shipping location) is required in order to achieve shipment by ground transportation. Supply shipments requiring priority delivery due to insufficient lead time for

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ground transportation shall be charged to the client at TestAmerica's cost. Alternatively, TestAmerica can ship the supplies via carrier of choice by the client using the client's shipping account.

Sample Storage/Disposal

TestAmerica will dispose of samples, sample extracts and digestates, at no additional cost to clients, 30 days after the final report is issued. Storage of samples and containers beyond this time frame may be available for an additional fee. Additional storage time may be available under normal circumstances for a fee starting at \$1 per container per month. Please contact your PM to inquire about availability and cost.

Subcontract Clause

TestAmerica reserves the right to perform the services at any laboratory in the TestAmerica network, unless the Client has specified a particular location for the work. In addition, TestAmerica reserves the right to subcontract services ordered by the Client to another laboratory or laboratories, if, in TestAmerica's sole judgment, it is reasonably necessary, appropriate or advisable to do so. TestAmerica will in no way be liable for any subcontracted services (outside the TestAmerica network) except for work performed at laboratories which have been audited and approved by TestAmerica.

TAT - Standard and Expedited

Data will be delivered at the proposed TAT in Business Days from Sample Receipt unless otherwise agreed upon.

Expedited turnaround time may be available and must be pre-approved by the laboratory. Expedited turnaround delivery is contingent upon meeting the agreed upon delivery date/time and numbers of samples. TestAmerica's expedited turnaround time surcharges are:

5 Business Days TAT = 30%
3 Business Days TAT = 60%
48 Hours TAT = 75%
24 Hours TAT = 100%

Taxes

Where reports are issued in or delivered to a state which assesses sales tax on TestAmerica's services, applicable sales taxes will be added to the invoice as required by law, unless an appropriate sales tax exemption form is on file with TestAmerica.

Terms and Conditions

This quotation is based solely upon TestAmerica's standard product (routine QA/QC, detection limits, deliverables and standard turnaround times) and noted exceptions herein. The discounts incorporated into the pricing are based upon the sample quantity, test method, and schedule quoted. Any deviations may impact pricing and/or the acceptance of work. Final acceptance of this work is contingent upon a mutually agreed Sample Delivery Schedule. All sales are subject to TestAmerica's Terms and Conditions unless alternative terms have been agreed to in writing. Submittal of samples will indicate acceptance of TestAmerica's Terms and Conditions and other requirements as set forth in this quotation.

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MBAS

Matrix	Method	Test Description	Analyte			
				RL	MDL	Units
Water	425.1	Methylene Blue Active Substances (MBAS)	Methylene Blue Active Substances	0.2	0.12	mg/l LAS

TESTAMERICA TERMS AND CONDITIONS OF SALE (Short Form)

Where a purchaser (Client) places an order for laboratory, consulting or sampling services from TestAmerica Laboratories, Inc., a Delaware corporation (referred to as "TestAmerica"), TestAmerica shall provide the ordered services pursuant to these Terms and Conditions, and the related Quotation or Price Schedule, or as agreed in a negotiated contract. In the absence of a written agreement to the contrary, the Order constitutes an acceptance by the Client of TestAmerica's offer to do business under these Terms and Conditions, and an agreement to be bound by these Terms and Conditions. No contrary or additional terms and conditions expressed in a Client's document shall be deemed to become a part of the contract created upon acceptance of these Terms and Conditions, unless accepted by TestAmerica in writing.

1. ORDERS AND RECEIPT OF SAMPLES

1.1 The Client may place the Order (i.e., specify a Scope of Work) either by submitting a purchase order to TestAmerica in writing or by telephone subsequently confirmed in writing, or by negotiated contract. Whichever option the Client selects for placing the Order, the Order shall not be valid unless it contains sufficient specification to enable TestAmerica to carry out the Client's requirements. In particular, samples must be accompanied by: a) adequate instruction on type of analysis requested, and b) complete written disclosure of the known or suspected presence of any hazardous substances, as defined by applicable federal or state law. Where any samples which were not accompanied by the required disclosure, cause interruptions in the lab's ability to process work due to contamination of instruments or work areas, the Client will be responsible for the costs of clean up and recovery.

1.2 The Client shall provide one week's advance notice of the sample delivery schedule, or any changes to the schedule, whenever possible. Upon timely delivery of samples, TestAmerica will use its best efforts to meet mutually agreed turnaround times. All turnaround times will be calculated from the point in time when TestAmerica has determined that it can proceed with defined work following receipt, inspection of samples, and resolution of any discrepancies in Chain-of-Custody forms and project guidance regarding work to be done (Sample Delivery Acceptance). In the event of any changes in the sample delivery schedule by the Client, prior to Sample Delivery Acceptance, TestAmerica reserves the right to modify its turnaround time commitment, to change the date upon which TestAmerica will accept samples, or refuse Sample Delivery Acceptance for the affected samples.

1.3 TestAmerica reserves the right, exercisable at any time, to refuse or revoke Sample Delivery Acceptance for any sample which in the sole judgment of TestAmerica: a) is of unsuitable volume; b) may pose a risk or become unsuitable for handling, transport, or processing for any health, safety, environmental or other reason, whether or not due to the presence in the sample of any hazardous substance and whether or not such presence has been disclosed to TestAmerica by the Client; or c) holding times cannot be met, due to passage of more than 48 hours from the time of sampling or 1/2 the holding time for the requested test, whichever is less.

1.4 Prior to Sample Delivery Acceptance, the entire risk of loss or damage to samples remains with the Client, except where TestAmerica provides courier services. In no event will TestAmerica have any responsibility or liability for the action or inaction of any carrier shipping or delivering any sample to or from TestAmerica's premises. Client is responsible to assure that any sample containing any hazardous substance which is to be delivered to TestAmerica's premises will be packaged, labeled, transported and delivered properly and in accordance with applicable laws.

2. PAYMENT TERMS

2.1 Services performed by TestAmerica will be in accordance with prices quoted and later confirmed in writing or as stated in the Price Schedule. Quoted prices do not include sales tax. Applicable sales tax will be added to invoices where required by law. Where requested services on a group of samples received and logged in together at the laboratory total less than \$50, there will be a minimum transaction charge of \$50 for the sample group, or as shown on any related quote from TestAmerica, at TestAmerica's discretion. An Environmental Management Fee of 5% of the invoice value will also be applied, at TestAmerica's discretion.

2.2 Invoices may be submitted to Client upon completion of any sample delivery group. Billing corrections must be requested within 30 days of invoice date. Payment in advance is required for all Clients except those whose credit has been established with TestAmerica. For Clients with approved credit, payment terms are net 30 days from the date of invoice by TestAmerica, unless alternative terms have been agreed in a separate

written agreement, and payment shall be made without retainage, and will not be contingent upon the receipt of funds from third parties. All overdue payments are subject to an additional interest and service charge of one and one half percent (1.5%) (or the maximum rate permissible by law, whichever is lesser) per month or portion thereof from the due date until the date of payment. All fees are charged or billed directly to the Client. The billing of a third party will not be accepted without a statement, signed by the third party, that acknowledges and accepts payment responsibility.

2.3 In the event Client fails to make timely payment of its invoices, TestAmerica reserves the right to pursue all appropriate remedies, including withdrawing certifications, suspending work and withholding delivery of data under this order without recourse. Client shall be responsible for all reasonable fees, expenses, and costs of collection including but not limited to, arbitrator's and attorney's fees. TestAmerica reserves the right to refuse to proceed with work at any time based upon an unfavorable Client credit report.

3. CHANGE ORDERS, TERMINATION

3.1 Changes to the Scope of Work, price, or result delivery date may be initiated by TestAmerica after Sample Delivery Acceptance due to any condition which conflicts with analytical, QA or other protocols warranted in these Terms and Conditions. TestAmerica will not proceed with such changes until an agreement with the Client is reached on the amount of any cost, schedule change or technical change to the Scope of Work, and such agreement is documented in writing.

3.2 Changes to the Scope of Work, including but not limited to increasing or decreasing the work, changing test and analysis specification, or acceleration in the performance of the work may be initiated by the Client after sample delivery acceptance. Such a change will be documented in writing and may result in a change in cost and turnaround time commitment. TestAmerica's acceptance of such changes is contingent upon technical feasibility and operational capacity.

3.3 Suspension or termination of all or any part of the work may be initiated by the Client. TestAmerica will be compensated consistent with Section 2 of these Terms and Conditions. TestAmerica will complete all work in progress and be paid in full for all work completed.

4. WARRANTIES AND LIABILITY

4.1 Where applicable, TestAmerica will use analytical methodologies which are in substantial conformity with published test methods. TestAmerica has implemented these methods in its Laboratory Quality Manuals and referenced Standard Operating Procedures and where the nature or composition of the sample requires it, TestAmerica reserves the right to deviate from these methodologies as necessary or appropriate, based on the reasonable judgment of TestAmerica, which deviations, if any, will be made on a basis consistent with recognized standards of the industry and/or TestAmerica's Laboratory Quality Manuals. Client may request that TestAmerica perform according to a mutually agreed Quality Assurance Project Plan (QAPP). In the event that samples arrive prior to agreement on a QAPP, TestAmerica will proceed with analyses under its standard Quality Manuals then in effect, and TestAmerica will not be responsible for any resampling or other charges if work must be repeated to comply with a subsequently finalized QAPP.

4.2 TestAmerica shall start preparation and/or analysis within holding times provided that Sample Delivery Acceptance occurs within 48 hours of sampling or 1/2 of the holding time for the test, whichever is less. Where resolution of inconsistencies leading to Sample Delivery Acceptance does not occur within this period, TestAmerica will use its best efforts to meet holding times and will proceed with the work provided that, in TestAmerica's judgment, the chain-of-custody or definition of the Scope of Work provide sufficient guidance. Reanalysis of samples to comply with TestAmerica's Quality Manuals will be deemed to have met holding times provided the initial analysis was performed within the applicable holding time. Where reanalysis demonstrates that sample matrix interference is the cause of failure to meet any Quality Manual requirements, the warranty will be deemed to have been met.

4.3 TestAmerica warrants that it possesses and maintains all licenses and certifications which are required to perform services under these Terms and Conditions provided that such requirements are specified in writing to TestAmerica prior to Sample Delivery Acceptance. TestAmerica will notify the Client in writing of any decertification or revocation of any license, or notice of either, which affects work in progress.

4.4 The warranty obligations set forth in Sections 4.1, 4.2 and 4.3 are the sole and exclusive warranties given by TestAmerica in connection with any services performed by TestAmerica or any Results generated from such services, and TestAmerica gives and makes NO OTHER REPRESENTATION OR WARRANTY OF ANY KIND, EXPRESS OR IMPLIED. No representative of TestAmerica is authorized to give or make any other representation or warranty or modify this warranty in any way.

4.5 Client's sole and exclusive remedy for the breach of warranty in connection with any services performed by TestAmerica, will be limited to repeating any services performed, contingent on the Client's providing, at the request of TestAmerica and at the Client's expense, additional sample(s) if necessary. Any reanalysis requested by the Client generating Results consistent with the original Results will be at the Client's expense. If resampling is necessary, TestAmerica's liability for resampling costs will be limited to actual cost or one hundred and fifty dollars (\$150) per sample, whichever is less.

4.6 TestAmerica's liability for any and all causes of action arising hereunder, whether based in contract, tort, warranty, negligence or otherwise, shall be limited to the lesser amount of compensation for the services performed or \$100,000. All claims, including those for negligence, shall be deemed waived unless suit thereon is filed within one year after TestAmerica's completion of the services. Under no circumstances, whether arising in contract, tort (including negligence), or otherwise, shall TestAmerica be responsible for loss of use, loss of profits, or for any special, indirect, incidental or consequential damages occasioned by the services performed or by application or use of the reports prepared.

4.7 In no event shall TestAmerica have any responsibility or liability to the Client for any failure or delay in performance by TestAmerica which results, directly or indirectly, in whole or in part, from any cause or circumstance beyond the reasonable control of TestAmerica. Such causes and circumstances shall include, but not be limited to, acts of God, acts of Client, acts or orders of any governmental authority, strikes or other labor disputes, natural disasters, accidents, wars, civil disturbances, equipment breakdown, matrix interference or unknown highly contaminated samples that impact instrument operation, unavailability of supplies from usual suppliers, difficulties or delays in transportation, mail or delivery services, or any other cause beyond TestAmerica's reasonable control.

5. RESULTS, WORK PRODUCT

5.1 Data or information provided to TestAmerica or generated by services performed under this agreement shall only become the property of the Client upon receipt in full by TestAmerica of payment for the whole Order. Ownership of any analytical method, QA/QC protocols, software programs or equipment developed by TestAmerica for performance of work will be retained by TestAmerica, and Client shall not disclose such information to any third party.

5.2 Data and sample materials provided by Client or at Client's request, and the result obtained by TestAmerica shall be held in confidence (unless such information is generally available to the public or is in the public domain or Client has failed to pay TestAmerica for all services rendered or is otherwise in breach of these Terms and Conditions), subject to any disclosure required by law or legal process.

5.3 Should the Results delivered by TestAmerica be used by the Client or Client's client, even though subsequently determined not to meet the warranties described in these Terms and Conditions, then the compensation will be adjusted based upon mutual agreement. In no case shall the Client unreasonably withhold TestAmerica's right to independently defend its data.

5.4 TestAmerica reserves the right to perform the services at any laboratory in the TestAmerica network, unless the Client has specified a particular location for the work. In addition, TestAmerica reserves the right to subcontract services ordered by the Client to another laboratory or laboratories, if, in TestAmerica's sole judgment, it is reasonably necessary, appropriate or advisable to do so. TestAmerica will in no way be liable for any subcontracted services (outside the TestAmerica network) except for work performed at laboratories which have been audited and approved by TestAmerica.

5.5 TestAmerica shall dispose of the Client's samples 30 days after the analytical report is issued, unless instructed to store them for an alternate period of time or to return such samples to the Client, in a manner consistent with U.S. Environmental Protection Agency regulations or other applicable federal, state or local requirements. Air samples in Summa canisters and tedlar bags are used and the containers cleaned immediately after testing, such that those samples are not retained. Longer storage periods may be requested and may be accommodated as space allows for

an additional charge. Any samples for projects that are canceled or not accepted, or for which return was requested, will be returned to the Client at his own expense. TestAmerica reserves the right to return to the Client any sample or unused portion of a sample that is not within TestAmerica's permitted capability or the capabilities of TestAmerica's designated waste disposal vendor(s). ALL DIOXIN, MIXED WASTE, AND RADIOACTIVE SAMPLES WILL BE RETURNED TO THE CLIENT, unless prior arrangements for disposal are made.

5.6 Unless a different time period is agreed to in any order under these Terms and Conditions, TestAmerica agrees to retain all records for five (5) years.

5.7 In the event that TestAmerica is required to respond to legal process related to services for Client, Client agrees to reimburse TestAmerica for hourly charges for personnel involved in the response and attorney fees reasonably incurred in obtaining advice concerning the response, preparation to testify, and appearances related to the legal process, travel and all reasonable expenses associated with the litigation. Additional consulting beyond that normally associated with lab reports will be billed at TestAmerica's current published rates.

6. INSURANCE

6.1 TestAmerica shall maintain in force during the performance of services under these Terms and Conditions, Workers' Compensation and Employer's Liability Insurance in accordance with the laws of the states having jurisdiction over TestAmerica's employees who are engaged in the performance of the work. TestAmerica shall also maintain during such period, Comprehensive General and Contractual Liability (limit of \$1,000,000 per occurrence/ \$2,000,000 aggregate), Comprehensive Automobile Liability, owned and hired, (\$1,000,000 combined single limit), and Professional/Pollution Liability Insurance (limit of \$5,000,000 per occurrence/aggregate).

7. AUDIT

7.1 Upon prior notice to TestAmerica, the Client may audit and inspect TestAmerica's records and accounts covering reimbursable costs related to work done for the Client, for a period of two (2) years after completion of the work. The purpose of any such audit shall be only for verification of such costs, and TestAmerica shall not be required to provide access to cost records where prices are expressed as fixed fees or published unit prices.

8. MISCELLANEOUS PROVISIONS

8.1 These Terms and Conditions, together with any additions or revisions which may be agreed to in writing by TestAmerica, embody the whole agreement of the parties and provide the only remedies available. There are no promises, terms, conditions, understandings, obligations or agreements other than those contained herein, and these Terms and Conditions shall supersede all previous communications, representations, or agreements, either verbal or written, between the Client and TestAmerica. These Terms and Conditions, and any transactions or agreements to which they apply, shall be governed both as to interpretation and performance by the laws of the state where TestAmerica's services are performed.

8.2 The invalidity or unenforceability, in whole or in part of any provision, term or condition hereof shall not affect in any way the validity or enforceability of the remainder to these Terms and Conditions, the intent of the parties being that the provisions be severable. The section headings of these Terms and Conditions are intended solely for convenient reference and shall not define, limit or affect in any way these Terms and Conditions or their interpretations. No waiver by either party of any provision, term or condition hereof or of any obligation of the other party hereunder shall constitute a waiver of any subsequent breach or other obligation.

8.3 The obligations, liabilities, and remedies of the parties, as provided herein, are exclusive and in lieu of any others available at law or in equity. Indemnifications, releases from liability and limitations of liability shall apply, notwithstanding the fault, negligence or strict liability of the party to be indemnified, released, or whose liability is limited, except to the extent of sole negligence or willful misconduct.

QC Summary

QC Item	Frequency	Criteria	Corrective Action
Initial Calibration (ICAL)	Prior to sample analysis or when CCV fails	$r^2 \geq 0.995$	Refer to SOP SA-QA-16
- Minimum 5-pt and a blank Second Source			
Initial Calibration Verification (ICV)	After each ICAL	$\%D \leq 10\%$	Refer to SOP SA-QA-16
Low-Level Continuing Calibration Verification (LL-CCV)	Daily; at the beginning of the analysis (Spiked at the RL)	$\%D \leq 25\%$	Refer to SOP SA-QA-16
Continuing Calibration Verification (CCV)	At the beginning and end of the analysis, and after every 10 samples (Spiked at the mid-level)	$\%D \leq 10\%$	Refer to SOP SA-QA-16
Calibration Blank (ICB/CCB)	After ICV and CCV	result $< 1/2$ RL	Terminate the analysis, correct problem and reanalyze affected samples.
Batch Definition	Analyzed together w/in 24-hr timeframe; not to exceed 20 field samples	Not Applicable	Not Applicable
Method Blank (MB)	One per batch	result $< 1/2$ RL	Refer to SOP SA-QA-17
Laboratory Control Sample (LCS)	One per batch	Within limits listed in the MLG	Refer to SOP SA-QA-17
Laboratory Control Sample Duplicate (LCSD)	One per batch, when insufficient volume is provided for MS/MSD	Within limits listed in the MLG	Refer to SOP SA-QA-17
Matrix Spike (MS)	One per batch	Within limits listed in the MLG	Refer to SOP SA-QA-17

QC Item	Frequency	Criteria	Corrective Action
Matrix Spike Duplicate (MSD)	One per batch	Within limits listed in the MLG	Refer to SOP SA-QA-17 Refer to SOP SA-QA-06
Initial Demonstration of Capability (IDOC)	Initially; Per analyst	Within limits listed in the MLG	Note: Unsupervised work must not begin until successful IDOC is obtained.
Continuing Demonstration of Capability (CDOC)	Annually; Per analyst	Within limits listed in the MLG	Refer to SOP SA-QA-06
Method Detection Limit (MDL)	Upon method/instrument set-up, and then quarterly thereafter	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
MDL Verification (MDLV)	Upon method/instrument set-up, and then quarterly thereafter Upon method/instrument set-up, and then quarterly thereafter	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07
Reporting Limit Verification (RLV)	Note: MDLV may be performed in lieu of RLV.	Refer to SOP SA-QA-07	Refer to SOP SA-QA-07

Summary
C-4
48h

912-354-7858:
Bernard Krtland

METHOD #: 425

Approved for NPDES (Issued 1971)

TITLE:

Methylene Blue Active Substances (MBAS)

ANALYTE:

MBAS Methylene Blue Active Substances

INSTRUMENTATION:

N/A

STORET No.

38260

Interference 7 Cl⁻ 7000 ppm

0.05 - 100 ppm LAS

(linear alkyl sulfonate)

48h hold's time.

1.0 Scope and Application

- 1.1 This method is applicable to the measurement of methylene blue active substances (MBAS) in drinking waters, surface waters, domestic and industrial wastes. It is not applicable to measurement of surfactant type materials in saline waters.
- 1.2 It is not possible to differentiate between linear alkyl sulfonate (LAS) and alkyl benzene sulfonate (ABS) or other isomers of these types of compounds. However, LAS has essentially replaced ABS on the surfactant market so that measurable surfactant materials will probably be LAS type materials.
- 1.3 The method is applicable over the range of 0.025 to 100 mg/L LAS.

2.0 Summary of Method

- 2.1 The dye, methylene blue, in aqueous solution reacts with anionic-type surface active materials to form a blue colored salt. The salt is extractable with chloroform and the intensity of color produced is proportional to the concentration of MBAS.

3.0 Comments

- 3.1 Materials other than man-made surface active agents which react with methylene blue are organically bound sulfates, sulfonates, carboxylates, phosphates, phenols, cyanates, thiocyanates and some inorganic ions such as nitrates and chlorides. However, the occurrence of these materials at interference levels is relatively rare and with the exception of chlorides may generally be disregarded.
- 3.2 Chlorides at concentration of about 1000 mg/L show a positive interference but the degree of interference has not been quantified. For this reason the method is not applicable to brine samples.
- 3.3 Naturally occurring organic materials that react with methylene blue are relatively insignificant. Except under highly unusual circumstances, measurements of MBAS in finished waters, surface waters and domestic sewages may be assumed to be accurate measurements of man-made surface active agents.

4.0 Precision and Accuracy

- 4.1 On a sample of filtered river water, spiked with 2.94 mg LAS/liter, 110 analysts obtained a mean of 2.98 mg/L with a standard deviation of ± 0.272 .
- 4.2 On a sample of tap water spiked with 0.48 mg LAS/liter, 110 analysts obtained a mean of 0.49 mg/L with a standard deviation of ± 0.048 .
- 4.3 On a sample of distilled water spiked with 0.27 mg LAS/liter, 110 analysts obtained a mean of 0.24 mg/L with a standard deviation of ± 0.036 .
- 4.4 Analytical Reference Service, Water Surfactant No. 3, Study No. 32, (1968).

5.0 References

- 5.1 The procedure to be used for this determination is found in:
Standard Methods for the Examination of Water and Wastewaters, 14th Edition, p 600, Method No. 512A (1975).
Annual Book of ASTM Standards, Part 31, "Water", Standard D 2330-68, Method A, p 494 (1976).

[Communication]

425.1

Collected — 500 mL ^{plastic} amber glass / ~~two for sample / preserved~~
 — No preservation / keep chilled
 — non-filtered

[bernard.kirkland@kfstamerica.com]

Surfactants, Anionic (Detergents)

DOC316.53.01138

Crystal Violet Method¹

Method 8028

(0.002 to 0.275 mg/L as LAS)

Scope and Application: For water, wastewater and seawater.

¹ Adapted from *Analytical Chemistry*, 38, 791 (1966).



Test preparation

How to use instrument-specific information

The *Instrument-specific information* table displays information that may vary from instrument to instrument. Select the spectrophotometer from the instrument column on the left. Read across to find the corresponding sample cells and adapters required to perform this test on the spectrophotometer.

Table 1 Instrument-specific information

Instrument	Sample cell	Cell orientation
DR 5000	2612602	Fill line faces user
DR 3900	2612602	Fill line faces user
DR 3800, DR 2800, DR 2700	2612602	Fill line faces right

Before starting the test:

Use benzene only in a well-ventilated area.

Benzene (D018) solutions are regulated as hazardous waste by the Federal RCRA. Do not pour these materials down the drain. Collect water that is saturated with benzene and benzene solutions for disposal with laboratory solvent wastes. Refer to the current MSDS for safe handling and disposal instructions.

To prevent water droplets from forming in the sample cells, use only dry sample cells and discard the first few mL of benzene. Additionally, it can help to transfer the liquid from the funnel to a sample cell, let it sit for a few seconds and decant to a second cell for reading.

Excessive shaking may cause an emulsion to form, which makes the phases separate more slowly. If this occurs, remove most of the water layer, then gently mix the contents of the funnel with a clean Teflon®-coated rod or other inert tool.

Spilled reagent will affect test accuracy and is hazardous to the skin and other materials.

Acetone may be used to clean benzene from glassware.

For best results, measure a reagent blank value for each new lot of reagent (follow the procedure using deionized water in place of the sample). Subtract the reagent blank value from the final results or enter the value as a reagent blank adjust for automatic subtraction.

In bright light conditions (e.g. direct sunlight) be sure to cover the cell compartment during measurements.

The Pour-Thru Cell cannot be used with this procedure.

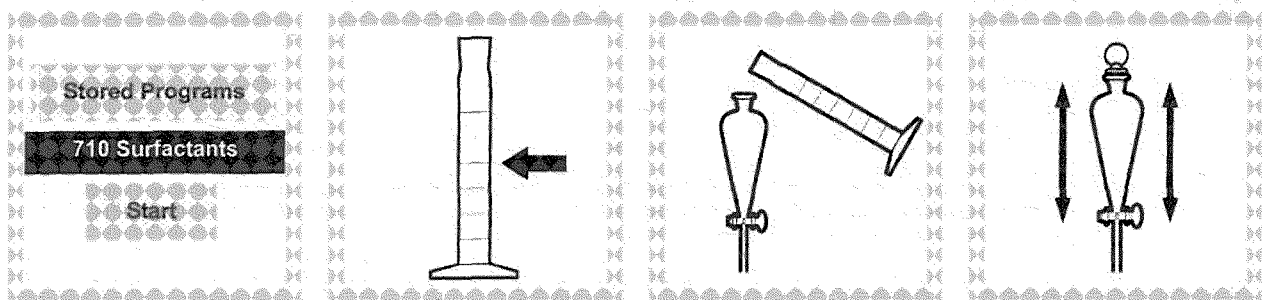
Surfactants, Anionic (Detergents)

Collect the following items:

Description	Quantity
Benzene, ACS	55 mL
Buffer Solution, sulfate-type	10 mL
Detergent Reagent Powder Pillows	1 pillow
Clippers, for opening powder pillows	1
Cylinder, graduated, 25-mL	1
Cylinder, graduated, 50-mL	1
Cylinder, graduated, 500-mL	1
Funnel, separatory, 500-mL	1
Sample Cells (see the <i>instrument-specific information table</i>)	2
Support Ring, 4-inch	1
Support, Ring Stand, 5 x 8 inch base	1

See *Consumables and replacement items* for reorder information.

Crystal Violet Method



1. Select the test.

Insert an adapter if required (*instrument-specific information table*).

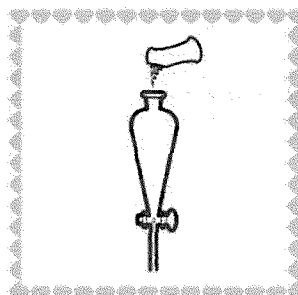
Refer to the user manual for orientation.

2. Fill a clean 500-mL graduated cylinder to the 300 mL mark with sample.

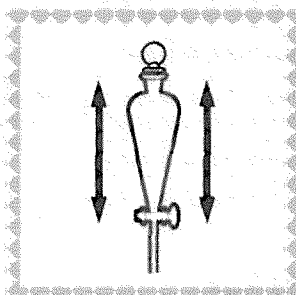
3. Pour the sample into a clean 500-mL separatory funnel.

4. Add 10 mL of Sulfate Buffer Solution. Stopper the funnel. Shake the funnel for five seconds.

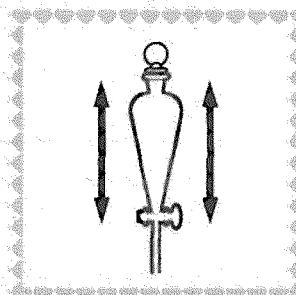
Crystal Violet Method (continued)



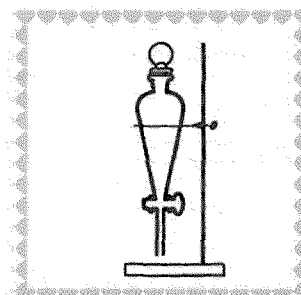
5. Add the contents of one Detergents Reagent Powder Pillow to the funnel.



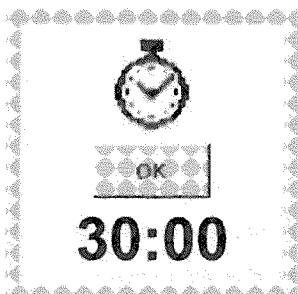
6. Stopper the funnel and shake until the powder dissolves completely. The powder will dissolve slowly.



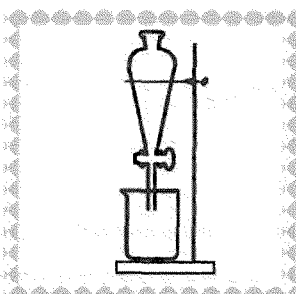
7. Add 30 mL of benzene to the funnel. Stopper the funnel and shake gently for one minute.



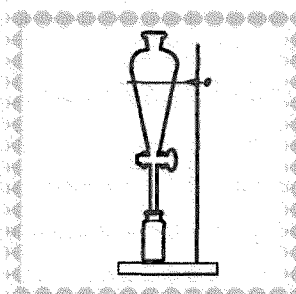
8. Place the separatory funnel in a support stand.



9. Start the instrument timer. A 30-minute reaction period will begin.

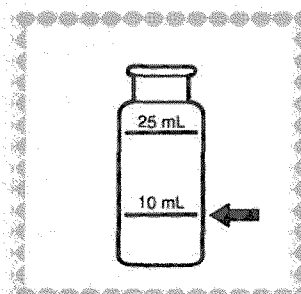


10. After the timer expires, remove the stopper and drain the bottom water layer. Discard this layer.

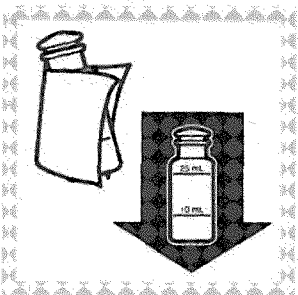


11. **Prepared Sample:** Drain the top benzene layer into a clean 25-mL sample cell. Stopper the cell.

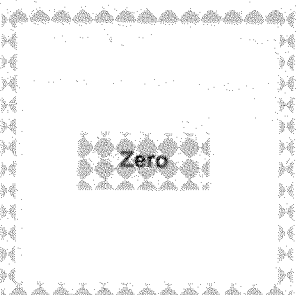
Do not filter the benzene layer before color measurement. Filtration removes the blue color.



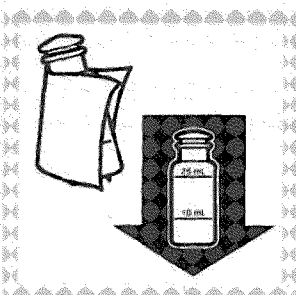
12. **Blank Preparation:** Fill another sample cell to the 10-mL mark with pure benzene. Stopper the cell.



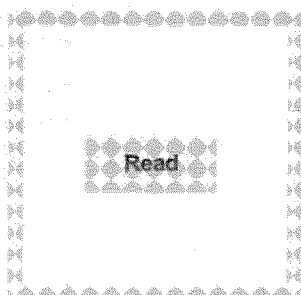
13. Wipe and insert the blank cell into the cell holder.



14. **ZERO** the instrument. The display will show: 0.000 mg/L LAS



15. Wipe and insert the sample cell into the cell holder.



16. **READ** the results in mg/L LAS.

Interferences

Table 2 Interfering substances

Interfering substance	Interference level
Chloride	High amounts of chloride, such as those levels found in brines and seawater, will cause low results.
Perchlorate ions	Interferes at all levels.
Periodate ions	Interferes at all levels.

Sample collection, preservation and storage

Collect samples in clean plastic or glass bottles. Analyze samples as soon as possible. Samples may be stored at least 24 hours by cooling to 4 °C (39 °F). Warm to room temperature before testing.

Accuracy check

Standard additions method (sample spike)

Required for accuracy check:

- Detergent Voluette® Ampule Standard, 60-mg/L LAS
- Ampule breaker
- TenSette Pipet, 0.1–1.0 mL and Tips

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.
2. Select standard additions from the instrument menu: **OPTIONS>MORE>STANDARD ADDITIONS**.
3. Accept the default values for standard concentration, sample volume and spike volumes. After the values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.
4. Open the standard solution ampule.
5. Prepare three sample spikes as follows. Measure 300 mL of sample into each of the three beakers. Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of standard, respectively, to each sample and mix thoroughly.
6. Follow the test procedure for each of the spiked samples, starting with the 0.1 mL sample spike. Measure each of the spiked samples in the instrument.
7. Select **GRAPH** to view the results. Select **IDEAL LINE** (or best-fit) to compare the standard addition results to the theoretical 100% recovery.

Standard solution method

Required for accuracy check:

- Detergent Voluette® Ampule Standard, 60-mg/L LAS
 - 1-L Class A volumetric flask
 - 3.0 mL Class A volumetric pipet and pipet bulb
1. Prepare a 0.180 mg/L LAS standard solution as follows: Pipet 3.0 mL of Detergent Standard, 60-mg/L as LAS, into a 1000-mL (1 liter) volumetric flask. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
 2. Follow the *Crystal Violet Method* test procedure.
 3. To adjust the calibration curve with the reading obtained with the standard solution, navigate to Standard Adjust in the software: **OPTIONS>MORE>STANDARD ADJUST**.
 4. Turn on the Standard Adjust option and accept the displayed concentration. If an alternate concentration is used, enter the concentration and adjust the curve to that value.

Method performance

Program	Standard	Precision 95% Confidence Limits of Distribution	Sensitivity Concentration change per 0.010 Abs change
710	0.180 mg/L LAS	0.172–0.188 mg/L LAS	0.002 mg/L LAS

Summary of method

Detergents, ABS (alkyl benzene sulfonate) or LAS (linear alkylate sulfonate) are determined by association with crystal violet dye and extraction of the ion-pair complex into benzene. Test results are measured at 605 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
Detergents Reagent Set, includes:	—	—	2446800
Benzene, ACS	40 mL	4 liters	1444017
Buffer Solution, sulfate-type	10 mL	500 mL	45249
Detergent Reagent Powder Pillows	1 pillow	25/pkg	100868

Required apparatus

Description	Quantity	Unit	Catalog number
Clippers, for opening powder pillows	1	each	96800
Cylinder, graduated, 25-mL	1	each	50840
Cylinder, graduated, 50-mL	1	each	50841
Cylinder, graduated, 500-mL	1	each	50849
Funnel, separatory, 500-mL	1	each	52049
Support Ring, 4-inch	1	each	58001
Support, Ring Stand, 5 x 8 inch base	1	each	56300

Surfactants, Anionic (Detergents)

Recommended standards

Description	Unit	Catalog number
Detergent Standard Solution, 10-mL Voluette® Ampule, 60-mg/L LAS	16/pkg	1427110

Optional reagents and apparatus

Description	Unit	Catalog number
Acetone, ACS	500 mL	1442949
Beaker, 600 mL	each	50052
Flask, volumetric, Class A, 1000 mL	each	1457453
Pipet filler bulb	each	1465100
Pipet, TenSette®, 0.1 to 1.0 mL	each	1970001
Pipet tips for TenSette Pipet 19700-01	50/pkg	2185696
Pipet, volumetric, Class A, 3.00 mL	each	1451503



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